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THE EFFECT OF PROCESSING PARAMETERS ON THE MORPHOLOGY OF AN IMMISCIBLE BINARY BLEND

by

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KEY WORDS

Polymer blend, processing, phase size, size distribution, coalescence, polypropylene, polycarbonate.

ABSTRACT

In this study an immiscible blend of polypropylene (PP) and polycarbonate (PC) was examined. Experiments were carried out in an internal mixer and the objective was to examine the influence of the time of mixing, the speed of rotation of the rotors as well as the effect of time under quiescent conditions on the size and size distribution of the minor phase. The morphology was studied by light and electron microscopy and the photographs were quantitatively analyzed by semi-automatic image analysis.

The effect of the time of mixing was studied for up to 20 minutes at three viscosity ratios for PC dispersed in PP. The most significant particle size deformation and disintegration processes take place within the first two minutes of mixing. After two minutes very little reduction in the size of the minor phase is observed up to 20 minutes mixing time, although some phase size/time dependence was observed at an intermediate viscosity ratio.

The influence of rotor speed was also studied and the results indicate that this blend is insensitive to changes in the speed of rotation of the rotor over a three to four-fold range.

The effect of time at 250°C under quiescent conditions was studied at 7% and 23% composition (volume fraction) for PP dispersed in PC and also for PC in PP. Coalescence coarsening was observed only for 23% PC in PP where the particle size was found to increase by 75% after approximately 90 minutes at temperature.

INTRODUCTION

Due to the deformable nature of the minor phase in immiscible blends a wide range of sizes and shapes can be generated during processing. Both the size and the shape of the minor phase can influence a multiplicity of physical properties including impact strength, [1,2], elongational properties [3] and permeability characteristics [4-6] to name a few.

During the processing of polymer blends several factors are especially important in determining the final size and shape of the minor phase: composition, viscosity ratio, interfacial tension, shear rate/shear stress, elasticity ratio and processing conditions (time of mixing, rate of rotation of rotor or screw, type of mixer). In our laboratory we have undertaken a series of studies aimed at understanding how both the size and shape of the minor phase may be controlled during processing as well as the effect of the morphology on final properties.

In a previous paper examining the influence of composition [7] it was shown that it had a marked effect on phase size especially as the region of dual phase continuity was approached (see Fig. 1). The blends were found to exhibit wide polydispersity in the size of the minor phase, a phenomena which is particularly pronounced at high concentration. This polydispersity was found to narrow with decreasing phase size. Changing the viscosity ratio has a significant effect on the phase size/composition relationship and a shift in the region of dual phase continuity was observed. The observed shift was confirmed by the viscosity data. The concentration at which dual-phase

continuity occurs does not correlate with model predictions based exclusively on viscosity ratio and composition.

In another study [8] the effect of the viscosity ratio (p) was studied in the region $p > 1$ and $p < 1$. It was found to have a significant effect on the morphology of the dispersed phase with the phase size increasing by a factor of 3 to 4 times from $p = 4.5$ to $p = 17.3$. Reduction in the size of the minor phase was achieved below $p = 1$ with the minimum particle size occurring at $p \approx 0.15$. The results for these systems indicate the presence of upper and lower limits of p beyond which deformation becomes difficult. This is shown to be similar in some respects to the behavior of Newtonian fluids in shear flow, although the upper limit extends beyond that observed in the Newtonian fluid studies. A linear relationship between the viscosity ratio and the torque ratio was shown to exist.

A preliminary study of the light scattering characteristics of these blends in the multiple scattering mode was also investigated in order to determine the suitability of various techniques for on-line characterization of both size and shape of the minor phase [9].

In polyamide/polyolefin blends the influence of processing conditions such as drawing and calandering on both the binary and compatibilized ternary blend systems were studied [10]. The shape of the minor phase was found to be very dependent on these processes. The influence of an ionomer compatibilizer on the size of the minor phase and the nature of the interactions at the

interface have also been studied [11].

The objective of this paper in the series is to consider the importance of processing parameters on the morphology of an immiscible binary blend of polypropylene/polycarbonate in an internal mixer. Parameters such as the time of mixing and the rate of rotation of the rotor and the influence of time under quiescent conditions are considered.

EXPERIMENTAL

(i) MATERIALS.

Two commercial grades of bisphenol-A polycarbonate, PC, and two of polypropylene, PP, were examined in this study. The polycarbonates are Merlon M-39, and M-50 obtained from the Mobay Corporation. The polypropylene samples are Pro-Fax 6301, and 6701 obtained from Himont. Their properties, including the number and weight average molecular weights, are summarized in Table 1. Throughout this paper polycarbonates M-39, and M-50 will be referred to as PC-1 and PC-2 respectively. Polypropylene 6301 and 6701 will be referred to as PP-1 and PP-2.

(ii) MIXING.

Prior to a typical mixing experiment the sample mixture was dried for 17 hours under vacuum at 90°C in order to minimize hydrolytic degradation of the polycarbonate during processing. The polypropylene was stabilized with 0.2% antioxidant. The materials were then melt blended in a Brabender mixing

chamber using the roller blade attachment normally used for viscous materials. The experiments were carried out under dry nitrogen. The volume of the material in the chamber was kept constant at 60 ml (calculated at 250°C) and the densities of polycarbonate and polypropylene at 250°C were 1.08 and 0.75 g/cc respectively. Blending conditions were maintained at 250°C and 50 RPM for 5 minutes.

A temperature of 250°C was chosen deliberately in order to minimize the thermal degradation of polypropylene. After blending, the melt was rapidly shaped into a block and cooled in ice water. The samples were stored at -25°C to eliminate any long term morphological change with time.

iii) MORPHOLOGICAL ANALYSIS.

Optical Microscopy - Polypropylene-rich samples for light microscopy were microtomed, exposed to methylene chloride to etch the polycarbonate, immersed in oil, and then photographed with high contrast film under bright field conditions. The microscope used was a Leitz/Wetzlar, Dialux 20.

Electron Microscopy - A scanning electron microscope (type JEOL 35 CF) was used to examine the polycarbonate-rich samples since the disperse phase tended to be of smaller size. The samples were microtomed to create a plane face using a microtome (Reicherg-Jung Supercut) equipped with a diamond knife. The samples were then subjected to decalin at 130°C for 15 minutes in order to dissolve the minor phase. Since the microtome does not necessarily cut the dispersed sphere at the widest point, a correction was applied in

order to obtain the true diameter [1].

Image Analysis - A semi-automatic method of image analysis was used to quantify the size of the minor phase. It consists of a unidirectional digitizing device that works as follows: as the crosshair of a magnifier connected to the mobile part of a linear variable differential tranformer (LVDT) is moved over the photomicrograph, the Ferrets diameter of a domain is recorded and stored in a microcomputer for the calculation of statistical parameters. Four fields of view and 200 diameters are considered for a given sample. The number average diameter, d_n, the volume average diameter (also known as the moment mean diameter), d_v, and full distribution curve were obtained in this way. Increasing the number of measurements to 400 or 600 did not significantly alter the results. The average error in the reported diameters is ±10%.

iv) RHEOLOGICAL ANALYSIS.

The Rheometrics Mechanical Spectrometer, RMS, Model 605, was used to measure the dynamic viscosity, η', and the storage modulus, G', as a function of frequency at 250°C. The experiments were carried out in the dynamic mode in parallel plate geometry at a strain of 10% and gap of 1.2 mm. The experiments were performed under dry nitrogen. The curves representing η' versus frequency are shown in Figure 1. The storage modulus data is reported elsewhere [8].

The torque values reported in the text were measured in the mixing

chamber at 250°C and at about 5 minutes mixing. Values for the pure materials are given in Table 1. The viscosity ratio is reported here as the ratio of the torque values for the pure components under the appropriate conditions. An excellent correlation was found previously [8] between the torque ratio (TR) and the viscosity ratio as measured on the RMS. Furthermore the torque ratio gives a measure of the viscosity ratio in the actual processing environment.

RESULTS AND DISCUSSION

Influence of the Time of Mixing on Dispersed Phase Size

Figure 2 shows the results of the dependence of phase size on the time of mixing at three different torque ratios. These results illustrate that the most important deformation/disintegration processes take place within the first two minutes of mixing, a period which is dominated by melting/liquefaction as shown by the torque time trace in Figure 3. Increasing the time of mixing from 2 to 20 min has little effect on the size of the dispersed phase for the high and low torque ratio blends. Electron micrographs are shown in Figure 4. At an intermediate torque ratio (TR = 7) some minor particle size reduction is observed with time. The significant effect of the torque ratio is evident from Figure 2 with a 6-fold difference in phase size observed for Blend-1 and Blend-3 at 5 min mixing time. The effect of torque ratio on phase size has been studied in more detail in a previous paper [8]. The size distribution of the minor phase also remains relatively unchanged for the high and low torque ratio blends as shown in Figure 5. At the intermediate torque ratio the size distribution narrows

slightly with mixing time. In all cases the log normal distribution is respected and a typical curve is shown in Figure 6.

Schreiber and Olguin [12] have studied the mixing behaviour of polyolefin/ elastomer blends in a Brabender mixing chamber and the sizes of the minor phase were determined by Quantimet analysis. In studying the relationship between phase size and energy input they found that the bulk of particle size reduction takes place very early in the mixing process. The dependence of phase size versus energy input then levels out very rapidly. Other authors have also noted that the time of mixing is not a critical variable [13]. The results in this paper indicate that a low dependence of phase size on mixing time exists over widely different torque ratios. At high torque ratio the very viscous dispersed phase resists deformation at longer times of mixing and at low torque ratio where finer dispersions are generated it becomes more difficult to deform small droplets. These concepts are in qualitative agreement with Taylor's theory [14, 15] for Newtonian fluids where the critical Weber number is proportional to the viscosity of the matrix, the shear rate and inversely proportional to the phase size and interfacial tension.

These results are significant in that they show the importance of the melting period in generating the final morphology of a blend during processing. These data also suggest that a detailed quantitative morphological study study of twin-screw extrusion where the screw design may be specifically constructed to modify shear in the melting zone would be a very worthwhile endeavour. The

literature contains very little information on this subject and work of this nature is currently underway in our laboratory. One particular paper of interest is that of Bartilla et al. [16] who have studied the effect of processing variables in 70:30 (by weight) polypropylene/ethylene propylene diene terpolymers (PP-EPDM) blends extruded on a twin-screw machine. These authors found that the presence of built in baffles (left-handed elements located downstream of the kneading blocks of the plastification zone) resulted in considerably more uniform and finer blends of EPDM in PP. Different metering zone geometries showed a perceptible but only minor effect on EPDM distribution. Compared to the melting zone, the differences achieved in the metering zone were slight. These results are confirmed to some extent in this study.

Influence of Rotor Speed

The influence of the rate of rotation of the rotors on the size of the dispersed phase is shown in Figure 7. Two blends at 7% volume fraction of minor phase were studied. One blend contained PC-1 dispersed in PP-1 (torque ratio = 7) while the other was the same blend with the matrix dispersed phase inverted (torque ratio = 0.14). Light micrographs are shown in Figure 8. These data clearly show that the rate of rotation of the rotors has little effect on phase size over a wide range of RPM. Figure 9 illustrates that this effect cannot be attributed to changes in the torque ratio with RPM since the torque ratio/RPM dependence for these systems is very low. If the measured torque is considered to be proportional to the shear stress and the RPM of the rotor proportional to the shear rate then Figure 4

approximately two fold increase in the apparent shear stress appears to have little influence on the morphology of this immiscible binary blend during melt mixing. This observation holds at both low and high torque ratio. Figure 10 shows that the distribution curves of minor phase size also show little variation with rotor speed. These results confirm a previous study where a two-fold change in shear stress was found to have little effect on phase size [8].

The effect of shear stress as considered in Taylor's equation for Newtonian fluids indicates that the phase size and shear stress are in inverse dependence. Wu [17] has shown in polyamide/rubber blends that changing shear stress resulted in observable differences in the size of the minor phase. By plotting the Weber number versus the viscosity ratio and hence taking into account the shear stress variable all his results fit on a single line master curve.

In other systems the effect of shear stress on phase size in immiscible binary blends has been less clear. Although large differences in shear stress clearly resulted in morphological changes [18] some authors [19] have indicated that varying the shear stress by a factor of two to three has little or no effect on the morphology. The results from this study suggest that both shear rate and shear stress do not appear to be continuous at the interface of this immiscible binary blend. The nature of the interface of these blends is shown in Figure 11 where fracture surfaces are shown for PC dispersed in PP. The dispersed spheres show a very sharp interface with no adhesion

whatsoever. Similar observations were made for PP in PC blends.

Coalescence Coarsening

In Table 2 the influence of time on phase size under quiescent conditions at 250°C is shown. In these experiments the samples were mixed for 5 min at 50 RPM and 250°C. The mixing was then stopped and the sample was held at temperature for the time indicated in Table 2. Coalescence coarsening was observed for 23% (volume fraction, ϕ) PC-1 in a matrix of PP-1. No coarsening effect was observed at 10% PC concentration. When the matrix and dispersed phase were inverted (PP-1 dispersed in PC-1), no coalescence coarsening was observed even at ϕ = 23% concentration.

The observation of coarsening in Table 2 shows that the number average diameter has increased by 75% after 85 minutes. The particle size distribution curves are shown in Figure 12. The size distribution (d_v/d_n) is not significantly changed and the log-normal distribution behavior is still respected (Figure 13). This result differs from the results of Jang et al. [20] where coalescence coarsening in systems was found to widen the size distribution.

Coarsening has not been studied extensively, but has been reported for several systems [20-23]. McMaster [21] using the transmission electron microscope observed coarsening in blends of styrene-acrylonitrile/polymethylmethacrylate blends. Jang, Uhlmann and Vander Sande [20] have observed coarsening for ethylene-propylene-diene terpolymer/polypropylene blends. Coarsening was

observed for 30% and 50% EPDM in PP. In general the initial process of coarsening was slow with more rapid coalescence occuring at later times. Coalescence coarsening is believed to represent the latter stages of phase separation. If high interfacial tension exists between two phases there will always be a tendency for the minor phase to minimize its surface free energy. Domain ripening for a system of discrete droplets dispersed in a matrix where the larger dispersed particles grow at the expense of the smaller ones is sometimes referred to as Ostwald ripening. A semi-quantitative analysis of the process was made by McMaster. The phenomenon was found to be related to: the interfacial tension, the volume fraction of the dispersed phase, the time of coalescence and the diffusion coefficient of the matrix.

In these experiments the importance of composition and matrix viscosity have been demonstrated. As previously mentioned, coalescence was observed at ϕ = 0.23 PC in PP-1 but not at ϕ = 0.07 PC composition. Also, no coalescence was observed for ϕ = 0.23 PP-1 in PC. The latter point indicates the importance of matrix viscosity and Table 2 shows that the zero shear viscosity of PC-1 is more than three times larger than that for PP-1. Thermal degradation during this long time at high temperature is shown to be minimal from the time-sweep studies from the RMS shown in Figure 14.

One of the reasons why this phenomena has not been studied is certainly related to the fact that long times as shown in this study are more than an order of magnitude longer than a typical processing cycle. During the course of our work however we examined higher composition samples (~50/50

mixtures). In samples of this type at or near the regions of dual-phase continuity significant morphological change was observed under quiescent conditions at very short time frames (~5min). The complexity of the morphological structures did not lend themselves to quantitative examination, but certainly indicates that coarsening may be an important practical consideration at or near the region of dual-phase continuity.

In our laboratory, work is being completed on the influence of temperature on the morphology of these systems. Work is also underway to examine the influence of processing parameters on phase size in compatibilized ternary blends to elucidate the role of good interfacial adhesion.

CONCLUSIONS

1 - Most of the deformation/disintegration processes occur in the first two minutes of mixing. Extending the time of mixing from 5 to 20 minutes has little influence on the minor phase size and distribution of PP/PC blends over a wide torque ratio range.

2 - A four-fold change in the apparent shear rate and doubling the apparent matrix shear stress has little effect on the dispersed phase size and distribution for both high and low torque ratio blends.

3 - Coalescence coarsening was observed for ϕ = 0.23 PC-1 in PP-1. This effect is found to be highly dependent on the concentration of the minor phase and the zero-shear viscosity of the matrix.

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FIGURES

Figure 1. η' versus ω for the neat polycarbonate and polypropylene samples at 250°C.

Figure 2. The influence of mixing time on minor phase size at three torque ratios (TR) for 7% PC in PP (volume fraction). Blend-1 is PC-2/PP-1, blend-2 is PC-1/PP-1 and blend-3 is PC-1/PP-2.

Figure 3. Typical torque/time trace for 7% PC-1 in PP-1 (volume fraction).

Figure 4. Scanning electron micrographs of 7% PC-1 in PP-1 (volume fraction) at a) 2 min and b) 20 min mixing time.

Figure 5. Minor phase size distribution curves for 7% PC in PP (volume fraction) showing the influence of mixing time at two torque ratios. a) blend PC-1/PP-1; b) blend PC-1/PP-2.

Figure 6. Typical log-normal distribution. Data illustrated is for sample at 20 min mixing time, 7% PC-1 in PP-2.

Figure 7. The effect of rotor speed on minor phase size at high and low torque ratio for 7% PC-1 in PP-1 and 7% PP-1 in PC-1. Blend PC-1/PP-1 has a torque ratio of 7 and blend PP-1/PC-1 has a torque ratio of 0.14.

Figure 8. Light micrographs of 7% PC-1 in PP-1 (volume fraction) showing the influence of rotor speed on phase size; a) 20 RPM, b) 90 RPM.

Figure 9. The dependence of the torque of pure PC-1 and pure PP-1 on rotor speed. The variation in torque ratio (TR) is indicated.

Figure 10. Minor phase size distribution curves showing influence of rotor speed at high and low torque ratio. a) blend of 7% PC-1/PP-1, torque ratio = 7; b) blend of 7% PP-1/PC-1, torque ratio = 0.14.

Figure 11. Fracture surface of 7% PC-1 in PP-1.

Figure 12. Minor phase size distribution curves showing the influence of coalescence time.

Figure 13. Log-normal distribution curves showing the influence of coalescence time.

Figure 14. 90 min time sweep of pure PC-1 and pure PP-1 at 250°C. The complex viscosity is plotted versus time.

Table 1. **CHARACTERIZATION OF POLYCARBONATE AND POLYPROPYLENE MATERIALS**

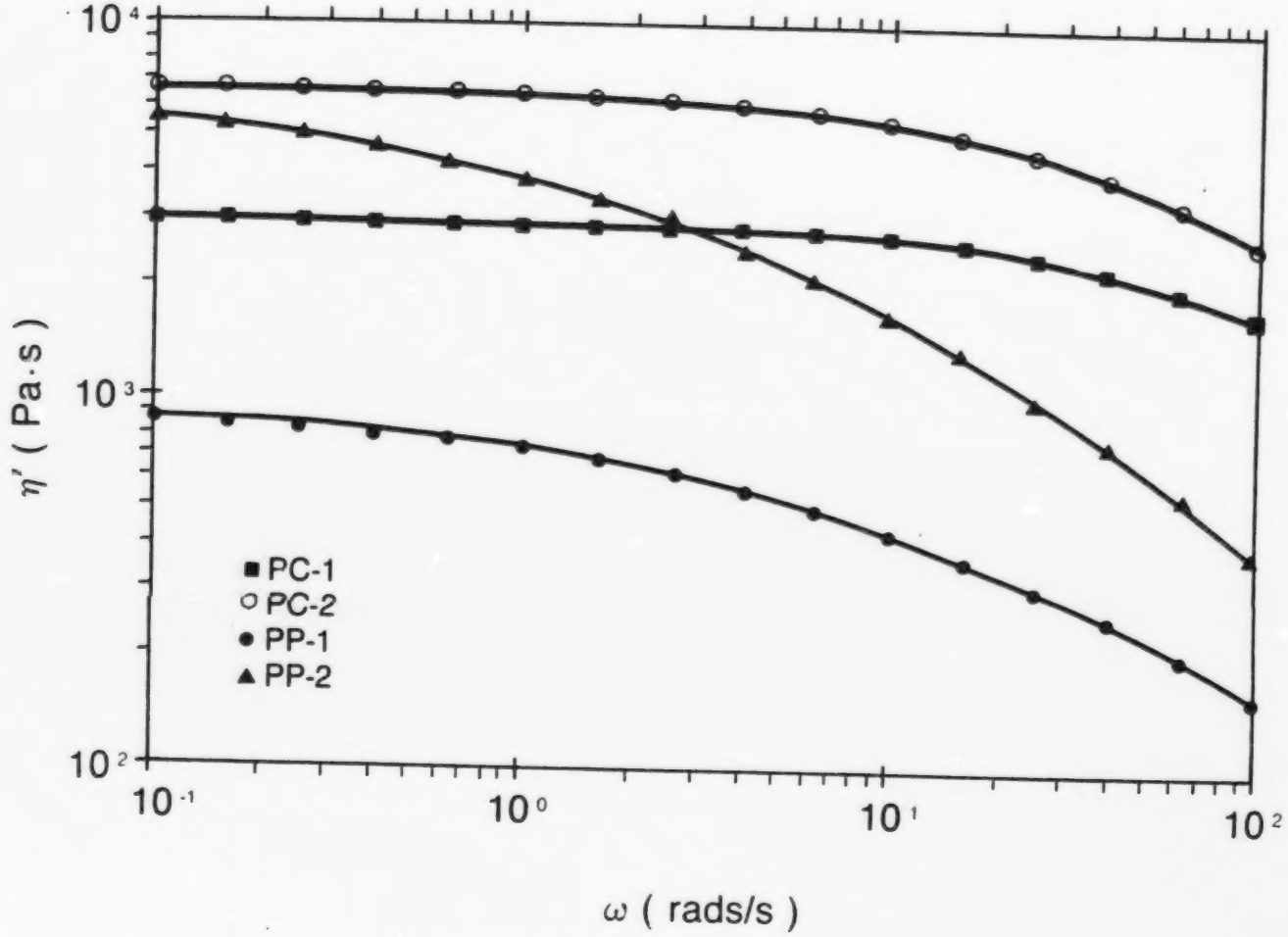
	[1] Melt Index (ASTM) (g/10 min)	[1] Density at 250°C (g/cc)	[2] Torque 5 min mixing 250°C (N·m)	[2] η' at 100 rad/s (Pa·s)	[1] $\bar{M}_n$	[1] $\bar{M}_w$
POLYCARBONATE						
(M-39) PC-1	18	1.08	26.9	1.79×10^3	12,300	26,500
(M-50) PC-2	4.5	1.08	49.8	2.76×10^3	16.500	36,300
POLYPROPYLENE						
(6301) PP-1	12	0.75	3.9	1.60×10^2	31,300	200,000
(6701) PP-2	0.8	0.75	12.7	3.93×10^2	72,000	470,000

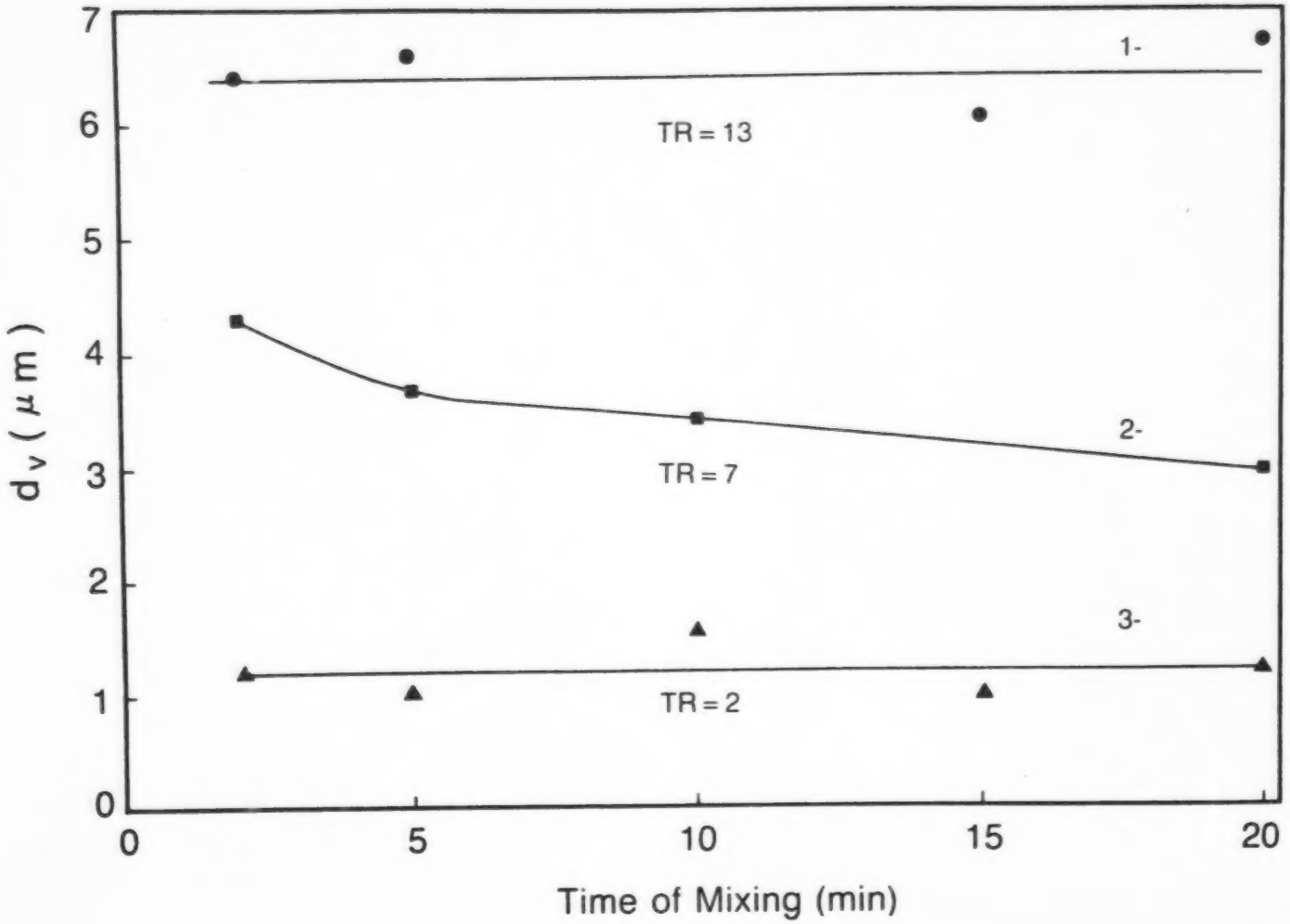
[1] obtained from suppliers.
[2] measured.

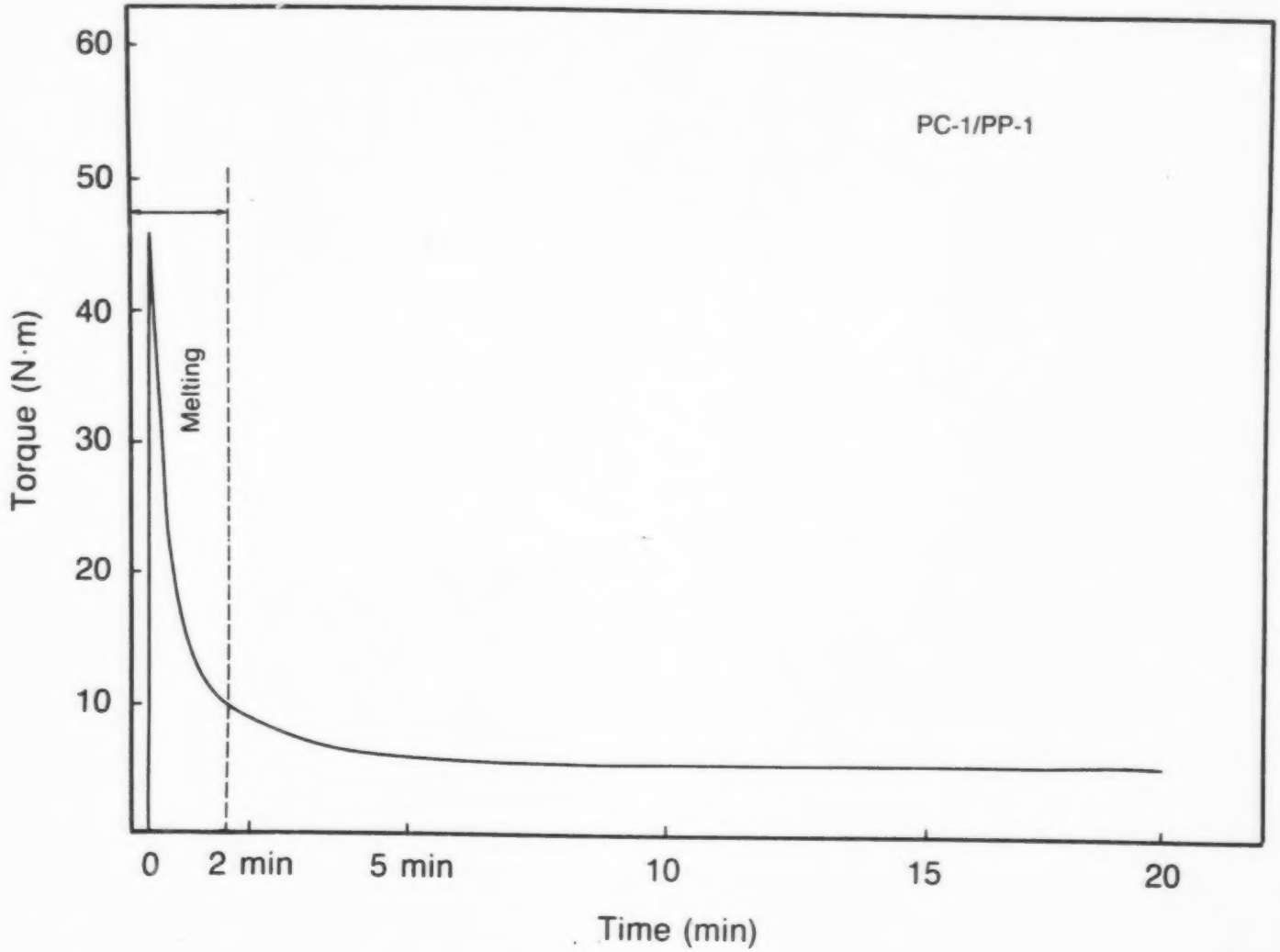
Table 2. COALESCENCE COARSENING

Time (min)	Volume Fraction of Minor Phase for Blends of PP-1 and PC-1	Zero Shear Viscosity of Matrix η_0 + (Pa•s)	d_n (μm)
0	0.23 PC-1	0.92×10^3	3.3
15			4.2
55			4.7
85			5.8
0	0.07 PC-1	0.92×10^3	2.3
85			2.4
0	0.23 PP-1	2.9×10^3	1.6
85			1.8

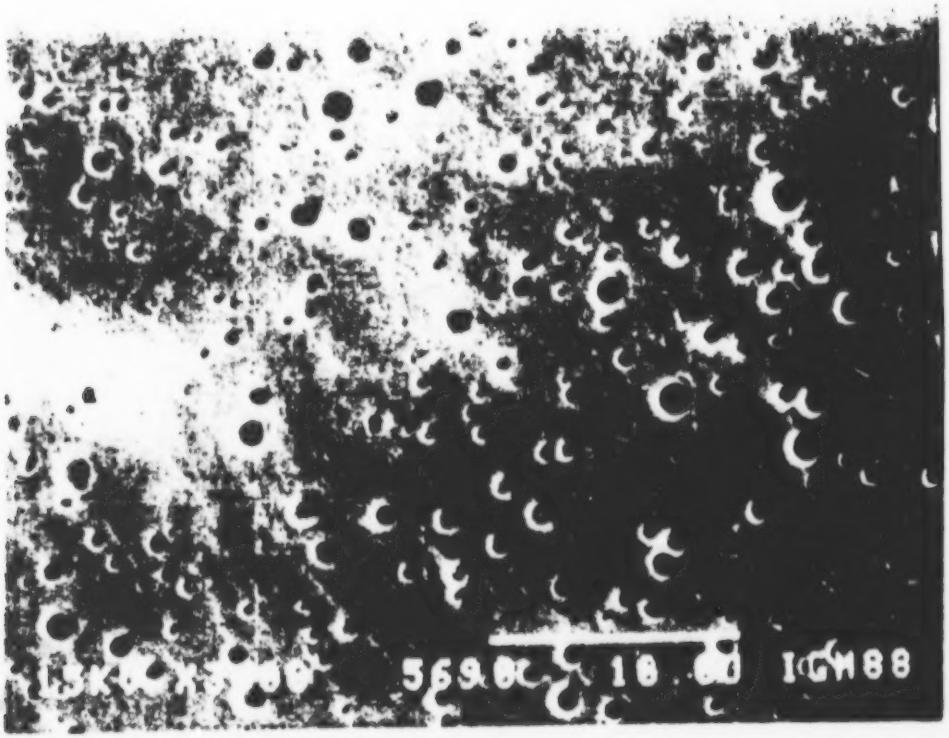
+ Calculated from RMS data by modified Carreau equation [25].


10 4
10 3
10 2
η' (Pa·s)
PC-1
PC-2
PP-1
PP-2
10 -1
10 0
10 1
10 2
ω (rads/s)


7
6
5
4
3
2
1
0
d_v (μ m)
1-
TR = 13
2-
TR = 7
3-
TR = 2
0
5
10
15
20
Time of Mixing (min)

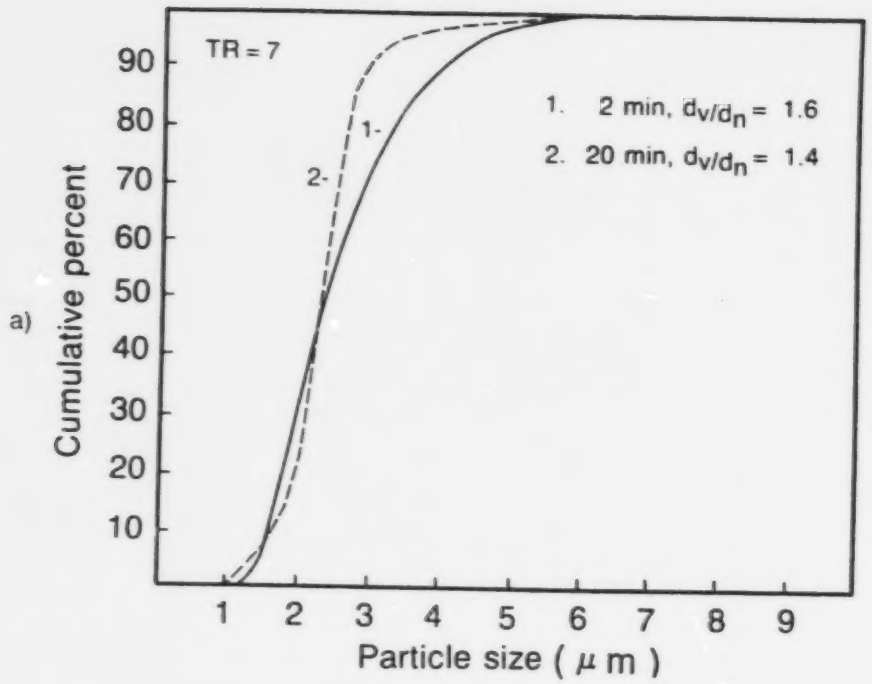

PC-1/PP-1
Torque (N·m)
60
50
40
30
20
10
Melting
0
2 min
5 min
10
15
20
Time (min)

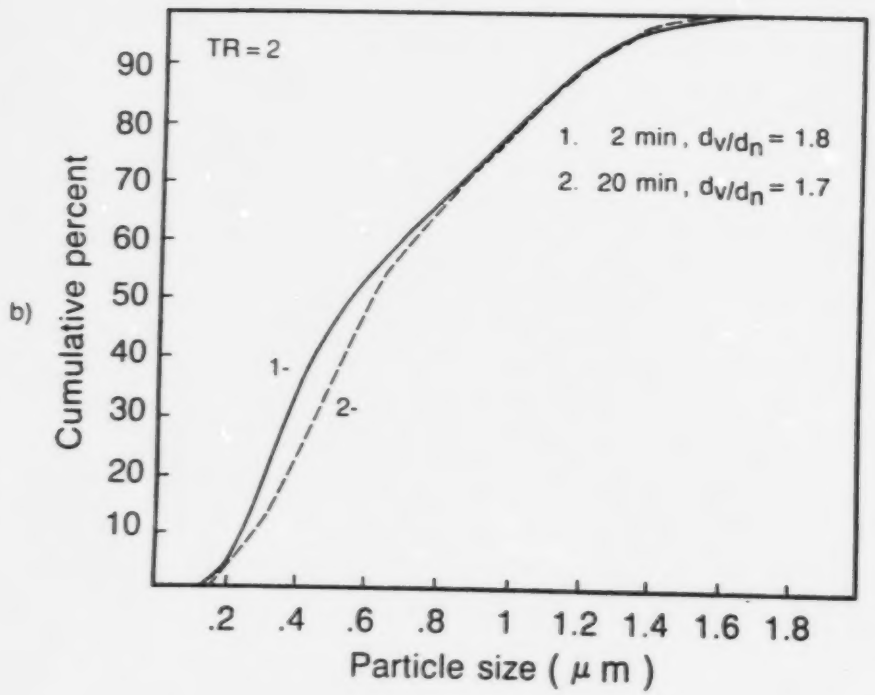
a)

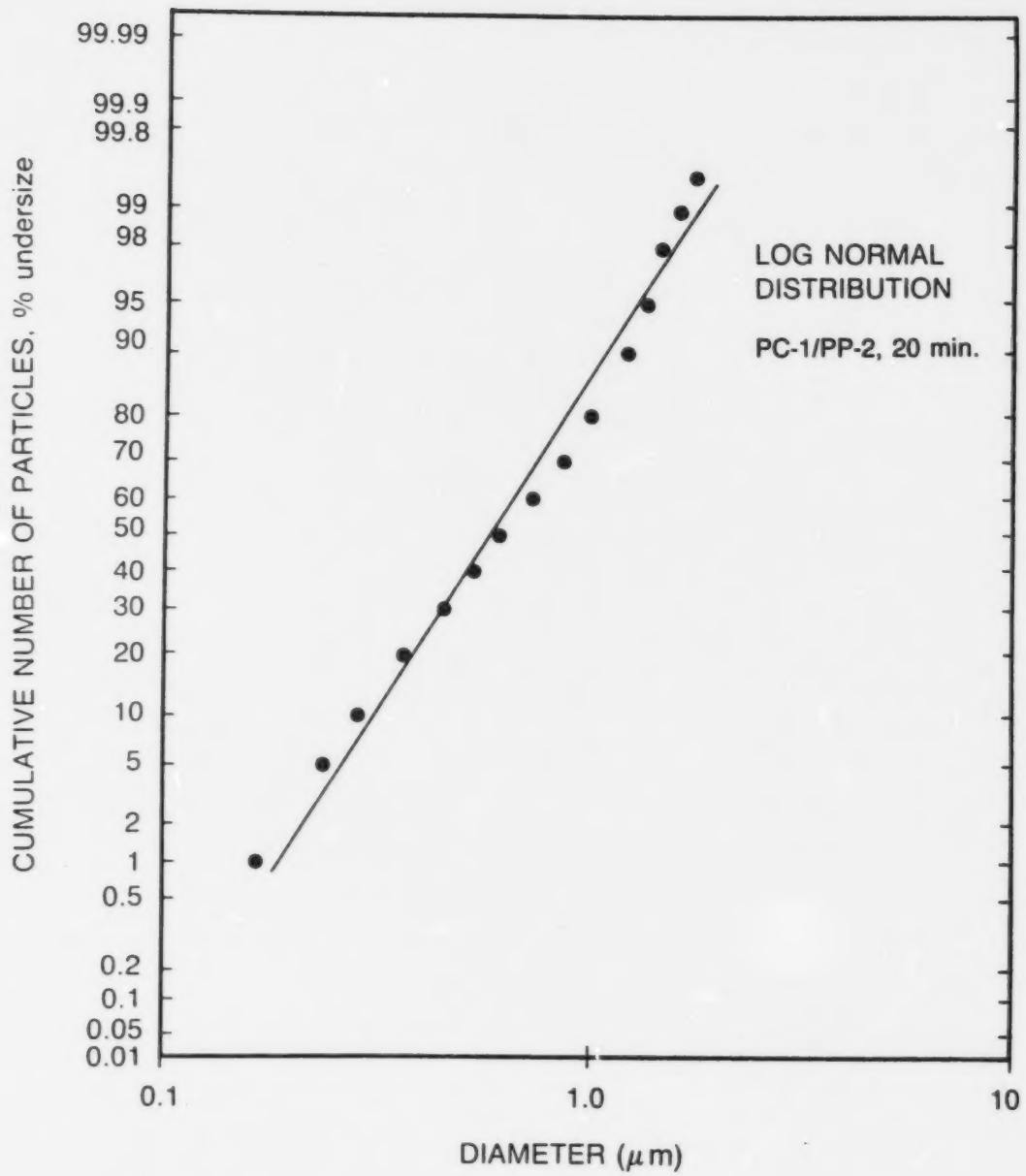


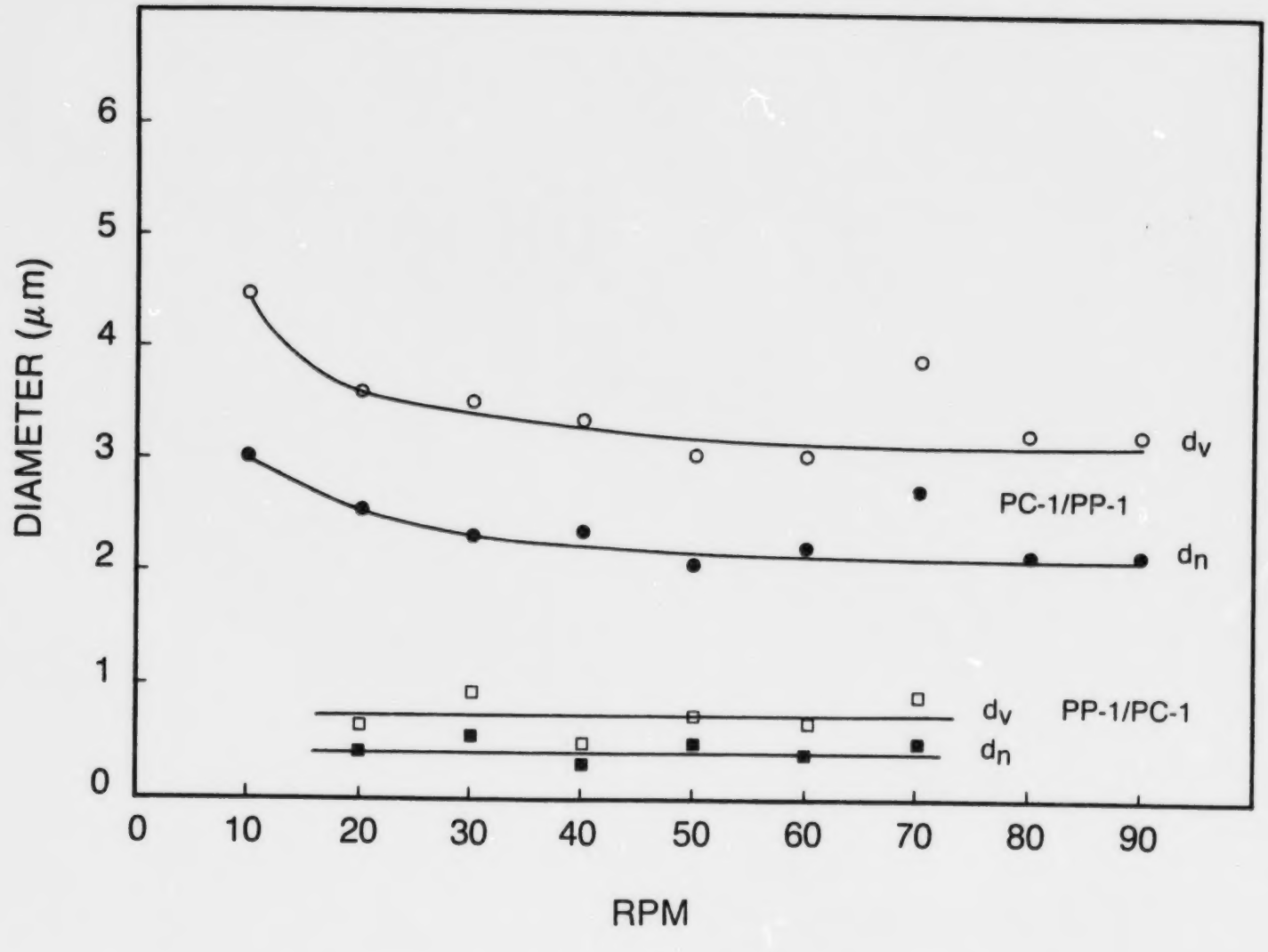
10µm

b)

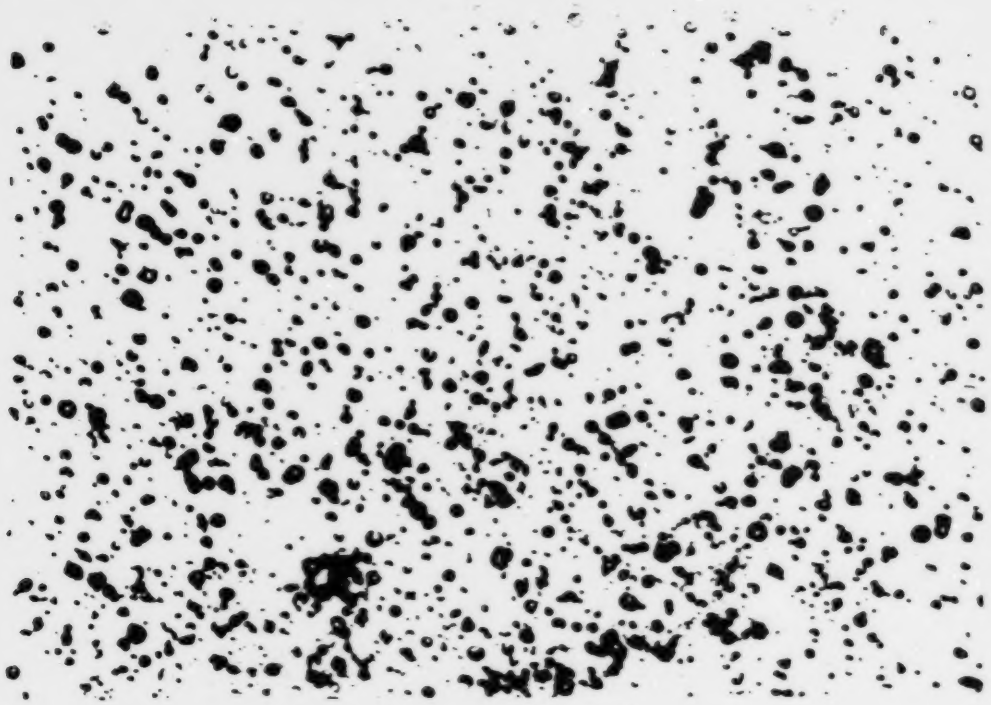

a)
TR = 7
1. 2 min, d_v/d_n = 1.6
2. 20 min, d_v/d_n = 1.4
1-
2-
Cumulative percent
90
80
70
60
50
40
30
20
10
1 2 3 4 5 6 7 8 9
Particle size (μ m)


b)
TR = 2
1. 2 min , d_v/d_n = 1.8
2. 20 min , d_v/d_n = 1.7
1-
2-
Cumulative percent
90
80
70
60
50
40
30
20
10
.2 .4 .6 .8 1 1.2 1.4 1.6 1.8
Particle size (μ m)


CUMULATIVE NUMBER OF PARTICLES, % undersize
99.99
99.9
99.8
99
98
95
90
80
70
60
50
40
30
20
10
5
2
1
0.5
0.2
0.1
0.05
0.01
0.1
1.0
10
DIAMETER (μm)
LOG NORMAL
DISTRIBUTION
PC-1/PP-2, 20 min.

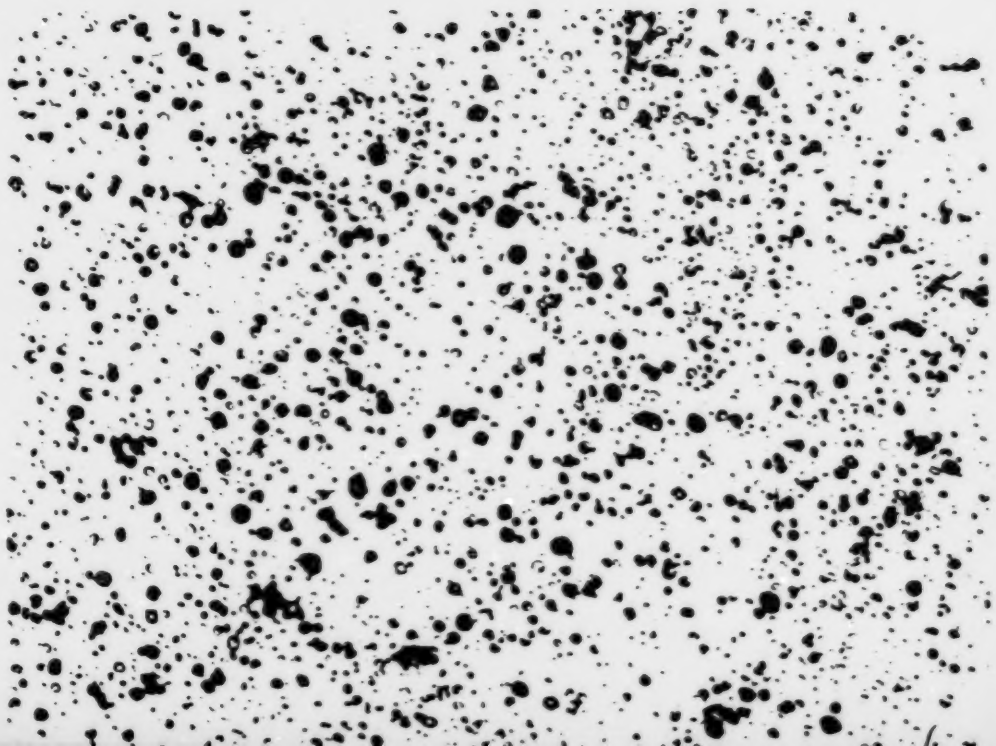

DIAMETER (μm)
6
5
4
3
2
1
0
0
10
20
30
40
50
60
70
80
90
RPM
d_v
PC-1/PP-1
d_n
d_v
PP-1/PC-1
d_n

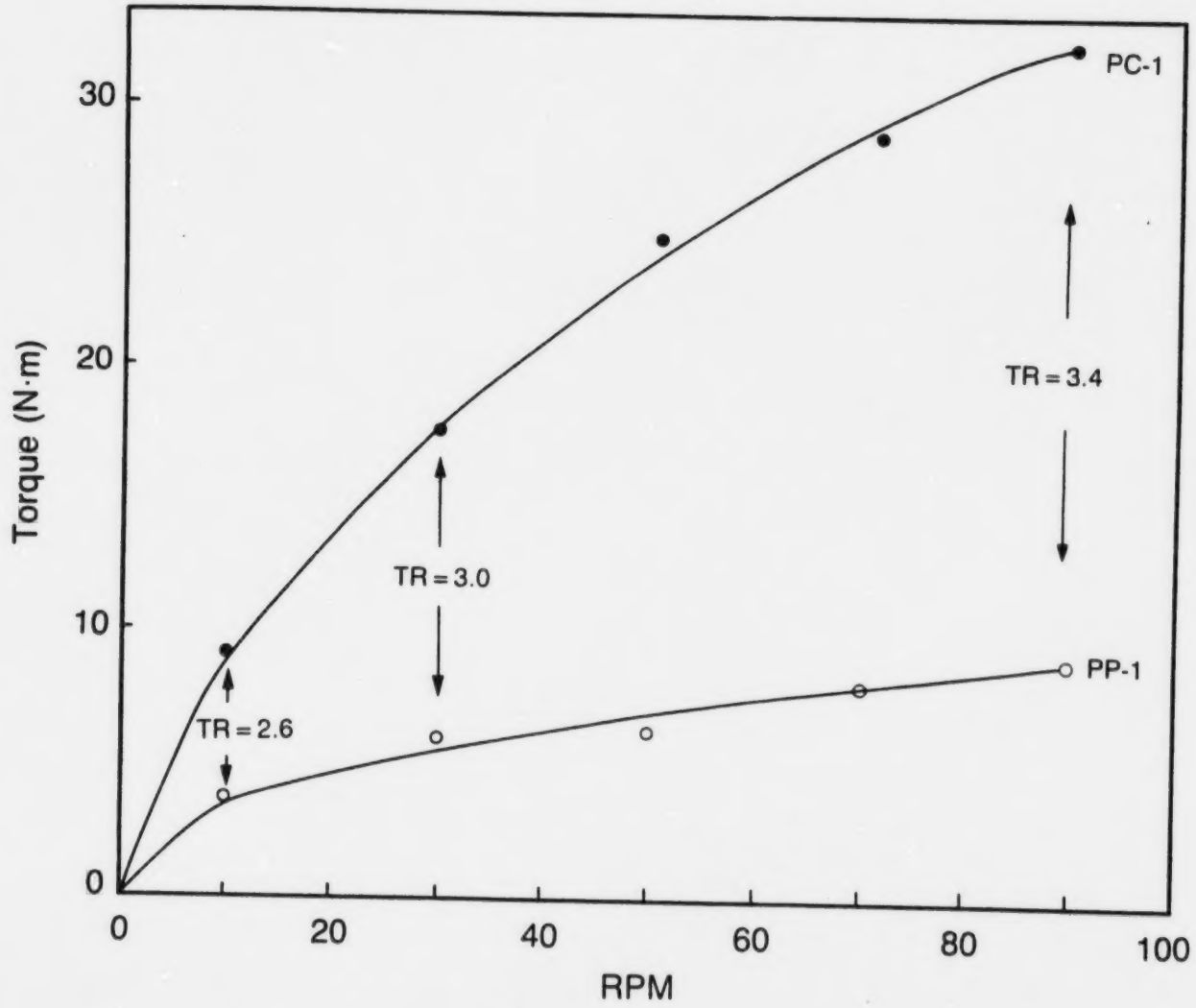
a)



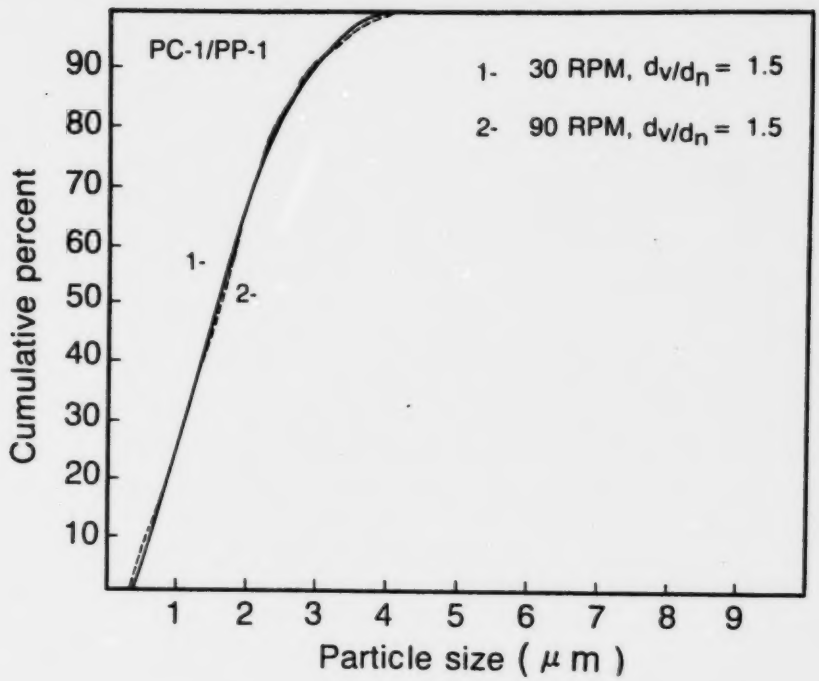
50 µm

b)

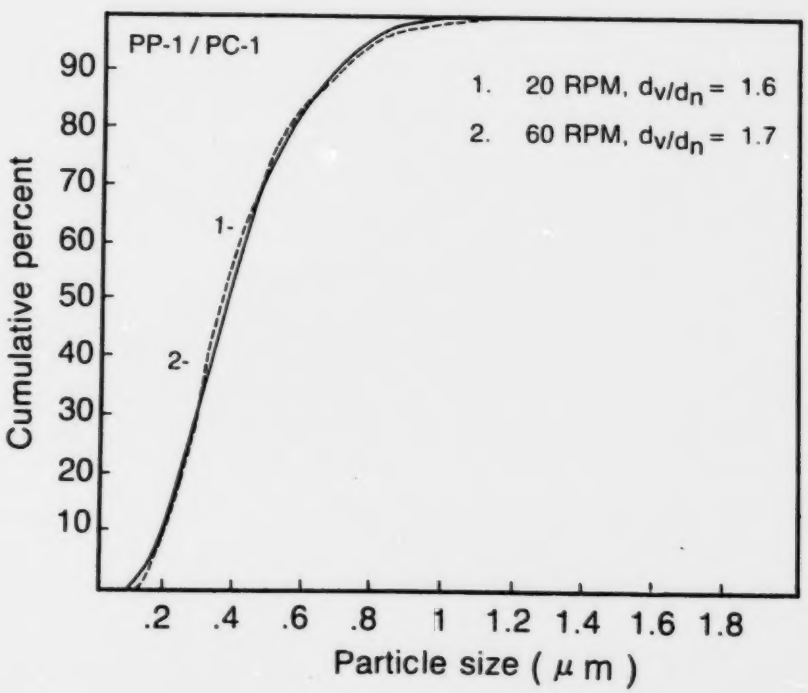


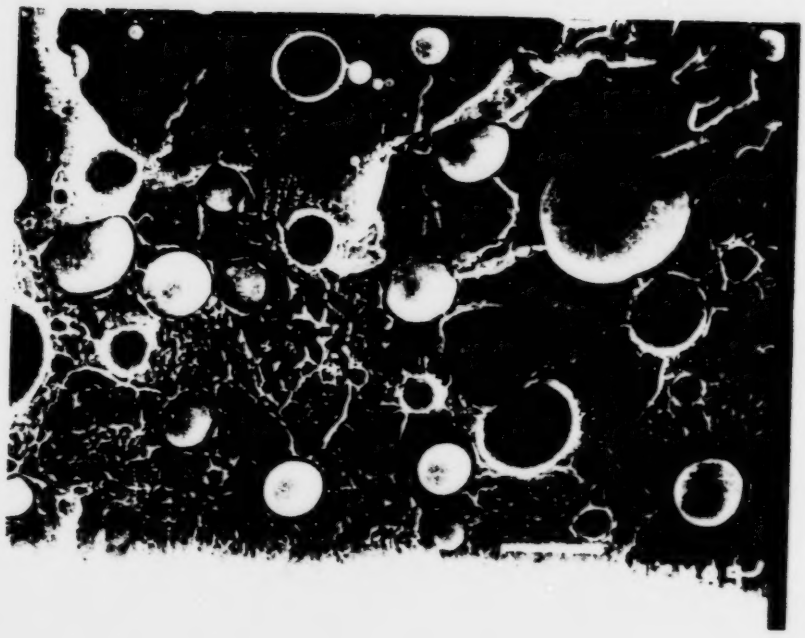

Torque (N·m)
30
20
10
0
0
20
40
60
80
100
RPM
PC-1
PP-1
TR = 3.4
TR = 3.0
TR = 2.6

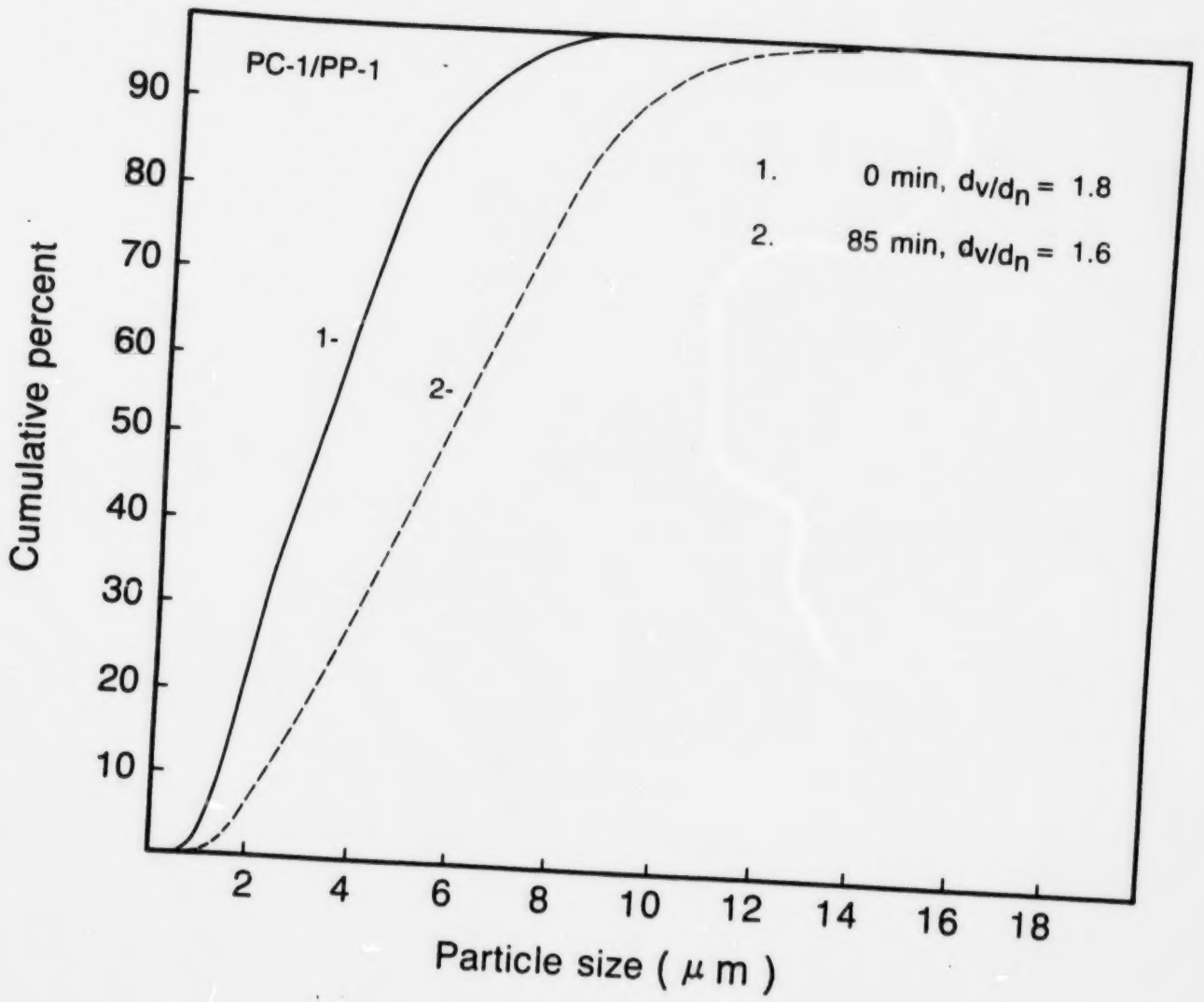
a)

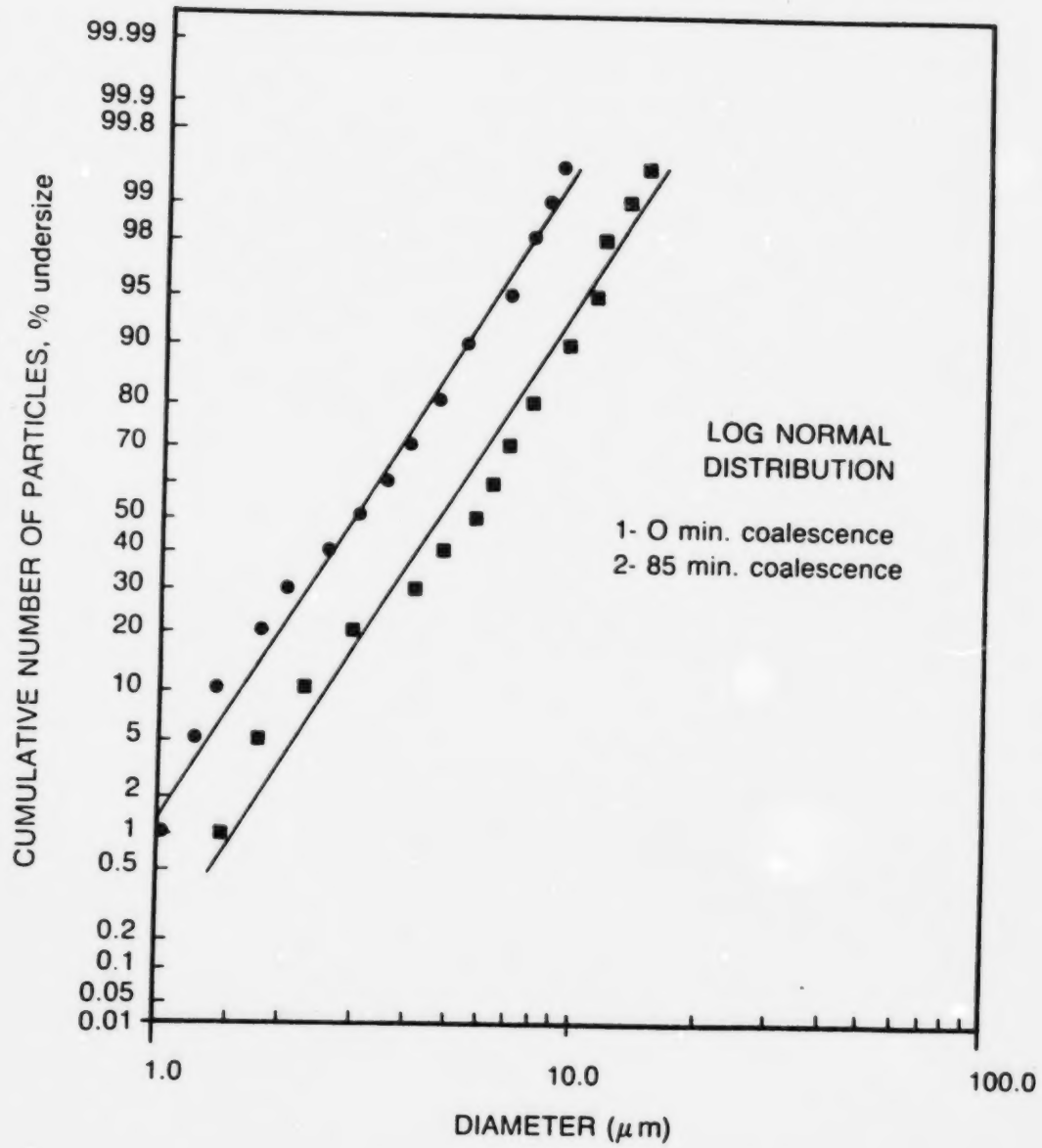

PC-1/PP-1
1- 30 RPM, d_v/d_n = 1.5
2- 90 RPM, d_v/d_n = 1.5
Cumulative percent
90
80
70
60
50
40
30
20
10
1-
2-
1 2 3 4 5 6 7 8 9
Particle size (μ m)

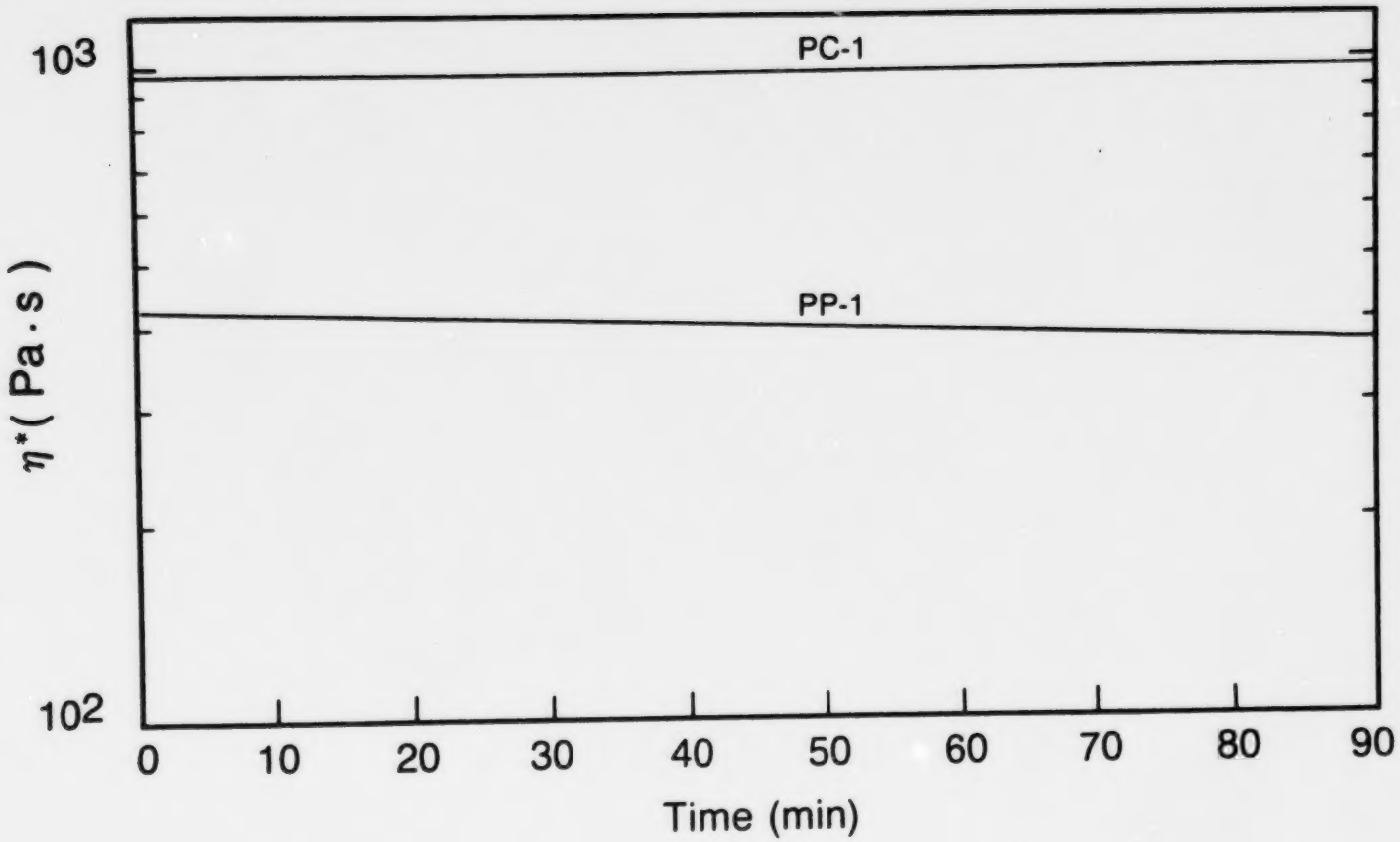
b)


PP-1 / PC-1
1. 20 RPM, d_v/d_n = 1.6
2. 60 RPM, d_v/d_n = 1.7
Cumulative percent
90
80
70
60
50
40
30
20
10
1-
2-
.2 .4 .6 .8 1 1.2 1.4 1.6 1.8
Particle size (μ m)




PC-1/PP-1
1. 0 min, d_v/d_n = 1.8
2. 85 min, d_v/d_n = 1.6
1-
2-
Cumulative percent
90
80
70
60
50
40
30
20
10
2
4
6
8
10
12
14
16
18
Particle size (μ m)


99.99
99.9
99.8
99
98
95
90
80
70
50
40
30
20
10
5
2
1
0.5
0.2
0.1
0.05
0.01
CUMULATIVE NUMBER OF PARTICLES, % undersize
LOG NORMAL
DISTRIBUTION
1- O min. coalescence
2- 85 min. coalescence
1.0
10.0
100.0
DIAMETER (μm)


PC-1
PP-1
10^3
10^2
η^*(Pa·s)
0
10
20
30
40
50
60
70
80
90
Time (min)